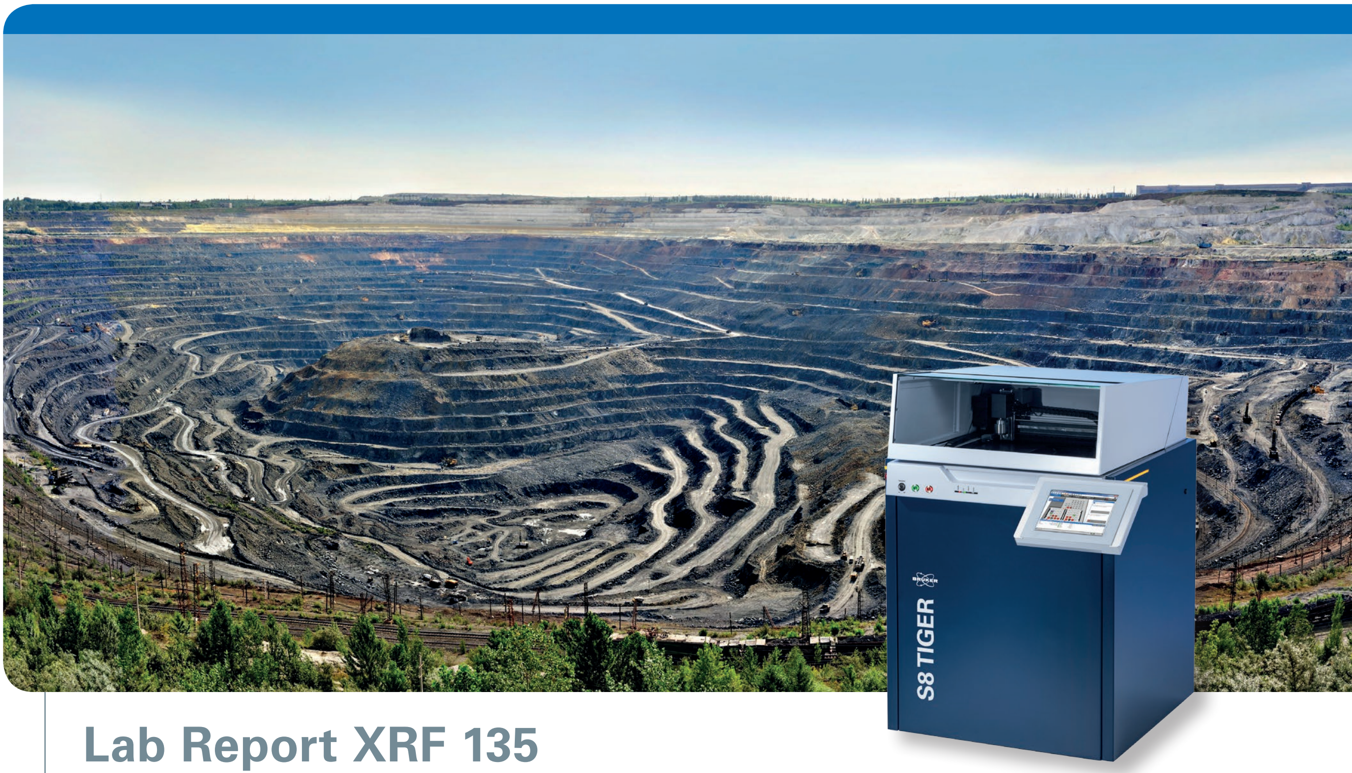


Lab Report XRF 135

S8 TIGER Series 2

High-Performance Trace Element Determination in Geological Samples

Introduction

The requirements for elemental analysis in geology are increasing with global issues such as the sustainable development of natural resources, protection of environment and people, information about erosion processes and the geochemical characterisation of regions, Today the powerful and cost-effective chemical profiling of many samples in a short time becomes even more important. Actual environmental and geological studies in research require the most reliable and accurate data. The industrial exploration and exploitation of minerals is demanding the lowest detection limits and highest sample throughput to realize cost-savings from effective process control.

High performance wavelength dispersive X-ray fluorescence (WDXRF) spectrometry has already been used for the determination of traces in geological samples. To fulfil today's requirements for quality data modern instruments must provide high elemental sensitivities to achieve low detec-tion limits and best spectral resolution in order to minimize line overlays. To cover the whole range of trace elements in a huge variety of different geological materials any analytical solution must be based on certified reference materials and a very effective handling of all matrix effects. This report outlines the performance of the wavelength dispersive X-ray fluorescence (WDXRF) spectrometer S8 TIGER for the determination of traces in geological samples.

Instrument

The S8 TIGER Series 2 is designed to serve all the needs of geological applications. It provides the optimum regarding detection limits, resolution and reliability for each element. It is delivered equipped with break-through HighSense technology, consisting of the high intensity X-ray tube in combination with the most compact beam path and low-noise HighSense counting electronics. The best excitation and highest intensity over the whole elemental range. The selection of the analyzer crystal LiF 220 and the 0.23° collimator produces the combination of choice for the analysis of traces starting from scandium up to uranium.

SampleCare[TM] of the S8 TIGER Series 2 offers unique benefits to users. Dust particles are often a problem for instruments analyzing samples prepared as pressed pellets. Sensitive spectrometer components are protected against damage by sample particles with the 4x protection by contamination shield.

The dust resistant design and sealed spectrometer cabinet also dedicates the S8 TIGER Series 2 for use in heavy industries like mining and ore processing. The simple instrument operation with with the TouchControl of the EZ Ergo configuration allows unskilled operators to learn and use the S8 TIGER Series 2 within short training times. Standard and Sample Preparation

One of the most important reasons for the use of XRF for geochemical characterization is the simple, fast and cost effective sample preparation. 10 – 12 grams of the dried sample is finely ground and mixed with grinding aid tablets in a ratio 5:1. Afterwards the material is pressed with a pressure of 20 tons within 20 s to form a stable pellet. This sample preparation technique is simple to establish into any laboratory environment and robust against contamination. The procedure is based on cost-effective equipment, safe non-hazardous chemicals and materials. To achieve highest sample throughput this procedure can be automated.

Measurement and Calibrations

Standards and samples were analyzed with the optimum excitation using the Rhodium tube with a voltage of 60 kV and a current of 67 mA. The analyzer crystal LiF220 ensures, that in combination with the 0.23° collimator, the best separation is achieved for adjacent elements.

The calibration was based on hundreds of international certified reference materials representing a huge variety of the most common geological materials. The standards were carefully selected to cover the typical concentration ranges for trace elements.

In total 22 elements were included within the measurement method for the quantitative analysis plus additional positions for the background modelling. Several major matrix elements were also measured to correct for their influences. As global matrix correction the Compton method was used based on the Rh Kα1 Compton line for most of the elements. The total measurement time with the S8 TIGER was less than 24 minutes.

Results

The calibration curves have been calculated for each element targeting precise and accurate trace analysis. The calibration ranges, measurement times and achieved detection limits are shown in table 1.

The detection limits were calculated according to

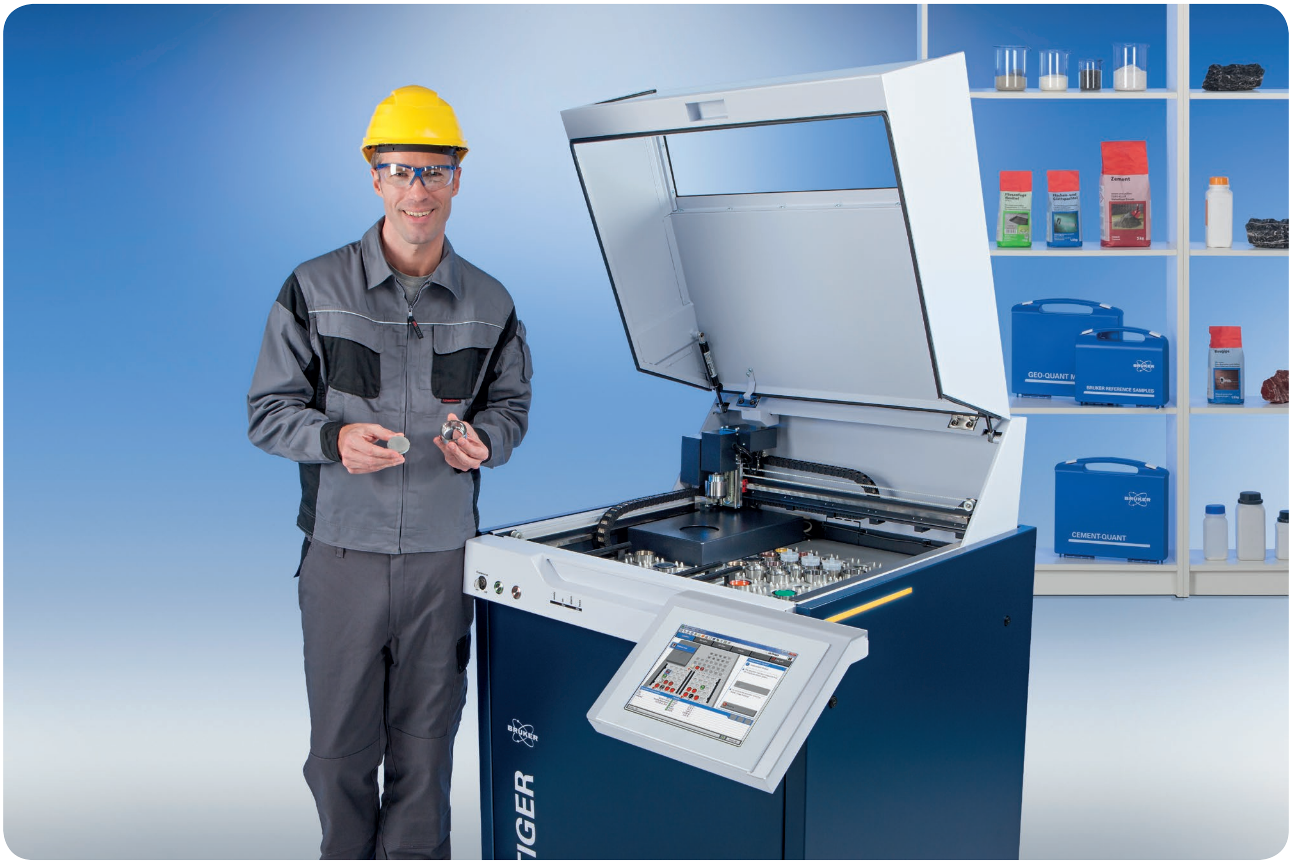
$$LLD = \frac{3}{m}\sqrt{\frac{I_b}{T_b}}$$

where m = sensitivity of analyte in kcps/mass%

I_b = background intensity for analyte in kcps

T_b = counting time in seconds at the background angle

The calibration is well suited to a wide range of geological materials. Adjustments of the measurement times to achieve even lower detection limits are possible to meet specific requirements.



WDXRF spectrometer S8 TIGER Series 2 for the analysis of trace elements in geological samples

The analytical accuracy of the trace element application was tested with two international certified reference materials (CRM) Mintek NIM-G and USGS W2A. Both materials were independent from the master calibration. The results are shown in table 2 and 3.

The data demonstrates the impressive analytical performance of the S8 TIGER Series 2 regarding accuracy and reliability. More than 22 elements are analysed accurately in less than 24 minutes (sample preparation included). The achieved precision outlines the robustness of the trace element application.

Conclusions

High performance trace analysis in geological materials is clearly a task for the wavelength dispersive X-ray fluorescence (WDXRF) spectrometer S8 TIGER Series 2. The precision and accuracy data presented clearly demonstrates that the instrumental setup ensures fast and reliable trace analysis. The simple and cost effective sample preparation in combination with the high analytical speed of the S8 TIGER Series 2 offers shortest time-to-result.

The integration of the S8 TIGER Series 2 into process and quality control for minerals and mining companies is seamless: SampleCare^{TM} of the S8 TIGER Series 2 and the dust resistant cabinet ensures the highest instrument up-time, TouchControl^{TM} offers the easiest operation and highest safety of fundamental data. Alongside analytical performance, simplicity and reliability are the most important demands for successful routine operation.

Table 1: Calibration Range and Measurement times of the geological trace calibration

Element Compound	LLD (100s, 3σ) [ppm]	Upper Calibration Range [ppm]	Analysis Time [s]
Sc	0.9	100	30
TiO_2	0.001 %	2.6 %	10
V	1.2	500	50
Cr	1.0	300	50
MnO	0.001 %	1 %	6
Fe_2O_3	0.001 %	20 %	4
Ni	0.7	2500	30
Cu	0.8	1000	30
Zn	0.5	3000	30
As	2.1	350	80
Rb	0.3	3500	20
Sr	0.3	1500	20
Y	0.4	150	20
Zr	0.3	1000	20
Nb	0.3	1000	20
Mo	0.2	150	30
Ba	3.8	2500	50
La	3.4	350	50
Ce	3.8	2500	50
Pb	0.9	2500	80
Th	0.8	1000	80
U	0.8	600	80

Table 2: Accuracy of the International Reference Sample Granite MINTEK NIM-G

Element / Compound	Certified Conc. [ppm]	XRF Conc. Mean Value 20 repetitions [ppm]	Abs. Std. Dev. 20 repetitions [ppm]
Sc	36	25	1
TiO_2	1.06 %	1.05 %	´0.001 %
V	260	235	1.5
Cr	92	86	1
MnO	0.16 %	0.16 %	0.005 %
Fe_2O_3	10.83 %	11.39 %	0.05 %
Ni	70	70	1.3
Cu	110	106	1.1
Zn	80	80	0.9
As	T	< 3	-
Rb	21	22	0.6
Sr	190	198	1.2
Y	23	22	0.6
Zr	100	96	0.8
Nb	8	6	0.6
Mo	T	< 1	-
Ba	170	156	2.8
La	10	11	2.4
Ce	23	18	2
Pb	9	6	0.9
Th	2	1.5	0.6
U	T	< 1	-

Link

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